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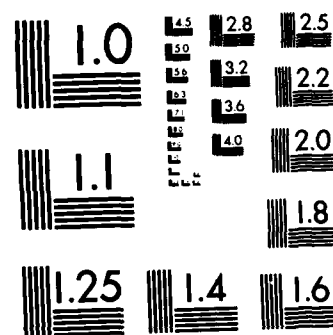
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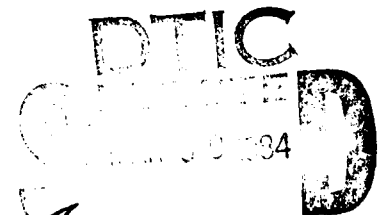
EFFECT OF INTERFACIAL BONDING ON THE STRENGTH OF
ADHESION OF ELASTOMERS III. INTERLINKING
BY MOLECULAR ENTANGLEMENTS

by

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found to be $15-25 \text{ J/m}^2$, generally about one-half of the work of fracture of the layers themselves, and consistent with the inferred density of interlinking molecular loops at the interface. The values were higher for higher densities, roughly in proportion, and for interlinking molecular strands of higher molecular weight, in accordance with the theory of Lake and Thomas. In the absence of interlinking the work of detachment was extremely small, only about 70 mJ/m^2 .

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SUMMARY

Polydimethylsiloxane (PDMS) networks have been formed by endlinking linear PDMS molecules. When a second layer is cast on top of a fully-gelled lower layer, the new molecules diffuse into the surface of the lower layer and form molecular loops ("entanglements") in the course of endlinking with themselves. The two layers are then joined only by these macromolecular loops. Measurements have been made of the work required to separate such layers under threshold conditions, i.e., at low rates, high temperatures, and, in some cases, in the swollen state. Values of the work of detachment have been found to be 15-25 J/m², generally about one-half of the work of fracture of the layers themselves, and consistent with the inferred density of interlinking molecular loops at the interface. The values were higher for higher densities, roughly in proportion, and for interlinking molecular strands of higher molecular weight, in accordance with the theory of Lake and Thomas. In the absence of interlinking the work of detachment was extremely small, only about 70 mJ/m².

INTRODUCTION

Measurements are reported here of the mechanical strength of molecular networks formed by the physical entanglement of elastomeric macromolecules. Previous studies have focussed on networks formed by interlinking macromolecules by covalent bonds (1-3). Such networks are relatively strong even under

threshold conditions, when dissipative processes are minimized. The work G_c of fracture per unit area torn through is then about 30-100 J/m², large in comparison with the theoretical value for a plane of C-C covalent bonds, about 1 J/m². This difference has been attributed by Lake and Thomas to the polymeric nature of the molecular strands comprising the network: many bonds must be stressed in order to break any strand (4). They concluded that the work of fracture is given by

$$G_c = K M_c^{\frac{1}{2}} \quad (1)$$

where M_c is the molecular weight of a network strand and K is a constant involving the density of the polymer, the effective mass, length and flexibility of a single main-chain bond, and the bond dissociation energy.

In Part I (5), two sheets of a crosslinked elastomer were placed in contact and joined by covalent chemical bonds. The work G_a of detachment under threshold conditions was found to increase in proportion to the density of interfacial bonds, from the low value characteristic of only Van der Waals' attractions up to the work G_c of fracture of the sheets themselves. Again, for a given density of interfacial interlinking the detachment work G_a was found to be greater when the network and interlinking strands were longer, in accordance with the concept of Lake and Thomas (5,6).

We now address the question: suppose two elastomeric sheets are connected together by molecules which loop around others in the opposite surface, forming permanent molecular

"entanglements", but are not connected directly by interlinking bonds, will the strength of adhesion be substantially different from the covalently-bonded case? The answer will be important for any quantitative treatment of the relationship between network structure and threshold strength.

The method employed for preparing pairs of elastomeric sheets joined only by "entanglements" was as follows. A layer of end-linkable polydimethylsiloxane (PDMS) linear polymer was poured as a flat sheet and then subjected to an end-linking (gelation) reaction using a tetrafunctional silane linking reagent (3,7). On top of the elastomeric PDMS sheet prepared in this way a second layer of PDMS polymer was poured and gelled in situ. Because the first layer was completely reacted, the second layer was unable to link with it directly. But, because of the relatively rapid interdiffusion of PDMS molecules (8), there will be a substantial penetration of the first layer by molecules of the second while they are in the process of end-linking to form the second elastomeric layer. Thus, the two layers will be joined by a permanent molecular entanglement wherever a molecule of the second layer loops around one of the first layer before end-linking is complete, Figure 1.

Values of the detachment work G_a per unit area of interface have been determined for sheets interlinked in this way. They are reported here for specimens prepared using PDMS of three different molecular weights, and they are compared with corresponding values for unentangled sheets, held together only by Van der Waals attractions.

EXPERIMENTAL DETAILS

(i) Preparation of polydimethylsiloxane (PDMS) networks

Networks were prepared as described elsewhere (3) from linear polydimethylsiloxanes having vinyl end groups. A tetrafunctional silane linking agent, tetrakisdimethylsiloxysilane, kindly supplied by Prof. C. W. Macosko, was mixed with the divinyl polymer, together with about 5 ppm of a Pt catalyst (9). The mixture was then degassed and cast as a thin sheet, about 1 mm thick, on a Teflon surface. Complete reaction, judged by the attainment of minimum swelling of the resultant gel in benzene, was achieved after 3 days at 70°C. Test sheets were therefore prepared by heating for 4 days at 70°C to ensure complete reaction.

Polymers having three different molecular weight ($\bar{M}_n = 11,500, 22,500$ and $36,000$ g/g-mole) were used for preparing networks. GPC data gave values for the ratio \bar{M}_w/\bar{M}_n of each polymer of approximately 2.0.

(ii) Preparation of interlinked layers

Samples were made by first casting one layer of endlinked PDMS, as described above, and then casting a second layer on top of it. In some cases the upper surface of the first layer was treated with 1-nonene and a small amount of a Pt catalyst (5 ppm) to saturate any residual SiH groups in the surface and thus prevent any possible chemical linking between the two layers. The top layer was then cast on in the usual way. No difference

was observed in the strength of adhesion for treated and untreated samples. It was concluded that no chemical linking occurred when the first layer was cured for 4 days at 70°C before the second layer was applied.

(iii) Measurement of the threshold work G_a of detachment

Measurements were made of the peel force required to separate two adhering layers, Figure 2. Samples were pre-swollen with m-xylene or silicone oil in some instances and immersed in a water bath at temperatures between 70°C and 90°C. The water effectively prevented evaporation of the swelling liquid. Only "symmetrical" samples were tested in the swollen state. "Symmetrical" samples are those in which both layers were made from PDMS of the same molecular weight, so that the crosslink density was the same and the degree of swelling would be equal. Values of the work G_a of detachment were then calculated from the measured peel force P per unit width:

$$G_a = 2\lambda_s^2 P \quad (2)$$

where λ_s is the linear swelling ratio. The term λ_s^2 in equation 2 accounts for the reduced number of network strands crossing the interface in a swollen specimen. For unswollen specimens, $\lambda_s = 1$.

Peeling was carried out in all cases at a rate of about 8 $\mu\text{m/s}$.

In order to study the strength of adhesion due to dispersion forces alone, thin sheets about 0.8 mm thick were cast and placed in contact with each other after cure. The upper surfaces of

cast sheets were smooth and shiny, and were brought into contact for these experiments. No difference was found in the measured peel force after 15 min and 24 h of contact. Therefore, 15 min was adopted as a standard contact time. Peeling separation was then carried out at room temperature at a rate of about 8 $\mu\text{m}/\text{s}$.

One pair of sheets was extracted with benzene at room temperature in an attempt to remove low-molecular-weight PDMS, inherent in PDMS materials. These samples gave the same (low) values of $\underline{G_a}$ as unextracted ones, 60-100 mJ/m^2 (Table 1). It is concluded that the strength of self-adhesion of PDMS materials under threshold conditions is extremely low, much lower than that observed previously for hydrocarbon elastomers, about 1 J/m^2 (5, 10). Possible reasons for this are discussed later.

RESULTS AND DISCUSSION

Values of $\underline{G_a}$ were measured for symmetrical interlinked specimens, both swollen and unswollen. They are given in Table 2. They were found to be in good agreement, when the factor λ_s^2 was employed to correct for changes in the interlinking density, and independent of the swelling liquid used. This agreement shows that threshold conditions had, indeed, been attained at the low rate of peel, 8 $\mu\text{m}/\text{s}$, and high temperatures, 70-90°C, used for these experiments.

Values of $\underline{G_a}$ for all of the interlinked samples are given in Table 3. They varied from 15 to 25 J/m^2 , depending upon

the molecular weights of the PDMS liquids used in preparing the top and bottom layers. When these molecular weights were both low, then the strength of adhesion was relatively low, and when they were higher, then the strength of adhesion was relatively high. This trend is consistent with previous results for the strength of adhesion of elastomer layers to each other (5) and to glass (10) and for their cohesive strengths also (2, 3, 6). In all cases, the work of fracture was greater when the network strands were longer, as predicted by Lake and Thomas (4). For the present materials, the network strand molecular weight is given by the molecular weight of the precursor polymer, to a first approximation.

In one case the upper layer could not be detached cleanly. It tore apart, requiring the input work $\underline{G_c}$ of cohesive rupture, instead of detaching. Molecules of the second layer had apparently penetrated the first layer to such an extent in this case that the interface was no longer the weakest plane. In all other cases, however, the work $\underline{G_a}$ of detachment was considerably smaller than the work $\underline{G_c}$ of cohesive rupture of either of the two adhering layers. Values of $\underline{G_c}$, taken from reference 3, are given in parentheses in Table 3, for comparison.

It is assumed that polymer molecules of the upper layer diffuse rapidly into the already-gelled lower layer and reach an equilibrium concentration in the surface regions quickly, before they undergo a significant amount of end-linking and gelation. Measurements of the equilibrium uptake of PDMS

liquids by PDMS gels have been reported previously (8). The results are given in Table 4 for the systems studied here. They range from 11 to 57 per cent uptake of the liquid by unit volume of the gel.

On comparing Tables 3 and 4, a general correlation is evident between the threshold work \underline{G}_a of detachment and the amount of PDMS liquid taken up at equilibrium, for each liquid. In Figure 3, values of \underline{G}_a are plotted against the volume uptake \underline{c} . They are seen to be described reasonably well by linear relations for each PDMS liquid forming the upper layer on later gelation. Thus, it appears that the strength of adhesion of the upper layer is approximately proportional to the amount of the precursor liquid absorbed by the lower layer, and hence to the number of interlinking strands. Moreover, the slopes of the linear relations shown in Figure 4 between \underline{G}_a and \underline{c} ; i.e., 60, 90 and 135 J/m²; are approximately in proportion to values of $\underline{M}_n^{1/2}$ for the PDMS liquid in the upper layer, which are in the ratio 65:90:115. Thus, the lengths of the interlinking strands also determine the strength of adhesion, in good quantitative agreement with the Lake-Thomas theory.

The horizontal broken lines in Figure 4 denote the work \underline{G}_c of cohesive rupture for each upper layer. By extrapolation in two cases, and by direct observation in the third, it is concluded that when the density of interlinking strands exceeds 35-50 per cent of those present in the lower layer then clean separation at the interface is no longer the clearly-preferred

mode of fracture. At this stage, the total density of chains crossing the interfacial plane will be about twice as large, 70-100 per cent, assuming that each pair of strands from the upper layer interlink successfully with a network mesh in the lower layer, so that the number of interlinking strands approaches that for a randomly-chosen plane within the upper or lower layer. It is not surprising, therefore, that cohesive rupture replaces interfacial separation at this point.

Finally, reference should be made to the extraordinarily low values obtained for adhesion between non-interlinked sheets, Table 1, in comparison with those obtained previously for the adhesion of sheets of hydrocarbon elastomers, about 1 J/m^2 (5). The present results are much closer to theoretical values for the maximum work of detachment due to Van der Waals' attractions alone, given by $2S$ where S is the surface energy of the elastomer layer. Thus, G_a is expected to fall in the range $40\text{-}80 \text{ mJm}^2$ on this basis, in good agreement with the present values for PDMS sheets.

However, it was found previously that other elastomeric materials adhere together much more strongly, and this was attributed to a generalization of the mechanism proposed by Lake and Thomas to account for their higher cohesive strength: that many bonds in a network strand must be stressed in order to break any one of them (5, 10). In the case of autohesion, the bond to be broken is the weak Van der Waals association at the interface, but the principle should still hold that the same

energy must be imparted to every bond in the molecular chain leading from the interface to the network itself, in order to break the association.

The question is, therefore: Are the present results representative of elastomeric materials in general, with the previous results for hydrocarbon elastomers anomalously high? Or, are the previous results representative of elastomeric materials in general, and the present results for PDMS layers anomalously low?

Extraction of PDMS sheets with benzene did not raise the level of adhesion significantly. Thus, there does not appear to be a liquid-like layer on the surface, responsible for low self-adhesion. On the other hand, it is extremely difficult to remove low-molecular-weight silicone fluids completely. The question raised above must therefore be considered unresolved, at present. It merits further study, in view of the large differences, by a factor of 10-20, between the two systems, and the theoretical and practical importance of the self-adhesion of elastomeric materials.

CONCLUSIONS

The main conclusion is that permanently "entangled" macromolecular loops appear to have about the same strength as covalently-bonded macromolecules of the same length. A second conclusion is that the work of rupture across a plane of such entanglements is roughly proportional to the inferred

density of entanglement interlinking. And, finally, the greater the molecular weight of the molecular strands comprising the loops, the greater appears to be the work of rupture, in accord with the theoretical treatment of Lake and Thomas for the cohesive strength of an elastomeric network. These conclusions have clear implications for the strength of molecular networks containing trapped entanglements.

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Table 1. Work G_a (mJ/m²) of detachment for
fully-cured PDMS layers, placed in contact

		Molecular weight \bar{M}_n of PDMS in upper layer		
		11,500	22,500	36,000
\bar{M}_n in lower layer	11,500	66 ± 3	60 ± 4	81 ± 2
	22,500	60 ± 4	80 ± 3	84 ± 2
	36,000	81 ± 2	84 ± 2	97 ± 4

Table 2. Threshold work $\underline{G_a}$ of detachment for
unswollen and swollen interlinked PDMS layers

Molecular weight $\underline{M_n}$ of PDMS in both layers

	11,500	22,500	36,000
Unswollen			
G_a (J/m ²)	14.5±0.8	19.5±1.5	24.9±1.4
Swollen in m-xylene			
λ_s	1.51	1.57	1.67
2P (N/m)	7.4±0.7	8.3±0.5	9.5±0.5
G_a (J/m ²) *	16.8±1.4	20.5±1.2	26.4±1.2
Swollen in silicone oil			
λ_s	1.19	1.24	1.30
2P (N/m)	10.5±1.3	12.7±0.9	14.7±0.8
G_a (J/m ²) *	14.7±1.7	19.6±1.4	24.8±1.3

$$* G_a = 2\lambda_s^2 P$$

Table 3. Threshold work G_a (J/m^2) of detachment for interlinked PDMS layers and threshold fracture energy G_c (J/m^2), in parentheses, for the layers themselves

		Molecular weight \bar{M}_n of PDMS in upper layer		
		11,500	22,500	36,000
\bar{M}_n in lower layer	11,500	15.5±0.4 (35)	17.1±0.4	18.9±0.5
	22,500	17.5±0.7	20.1±0.4 (44)	22.7±0.5
	36,000	(cohesive rupture)	24.6±0.6	25.6±0.4 (48)

Table 4. Volume of PDMS liquid taken up at equilibrium by unit volume of PDMS gels (taken from reference 8).

		Molecular weight \bar{M}_n of PDMS liquid		
		11,500	22,500	36,000
\bar{M}_n of PDMS gel	11,500	0.271	0.149	0.109
	22,500	0.375	0.220	0.162
	36,000	0.572	0.283	0.211

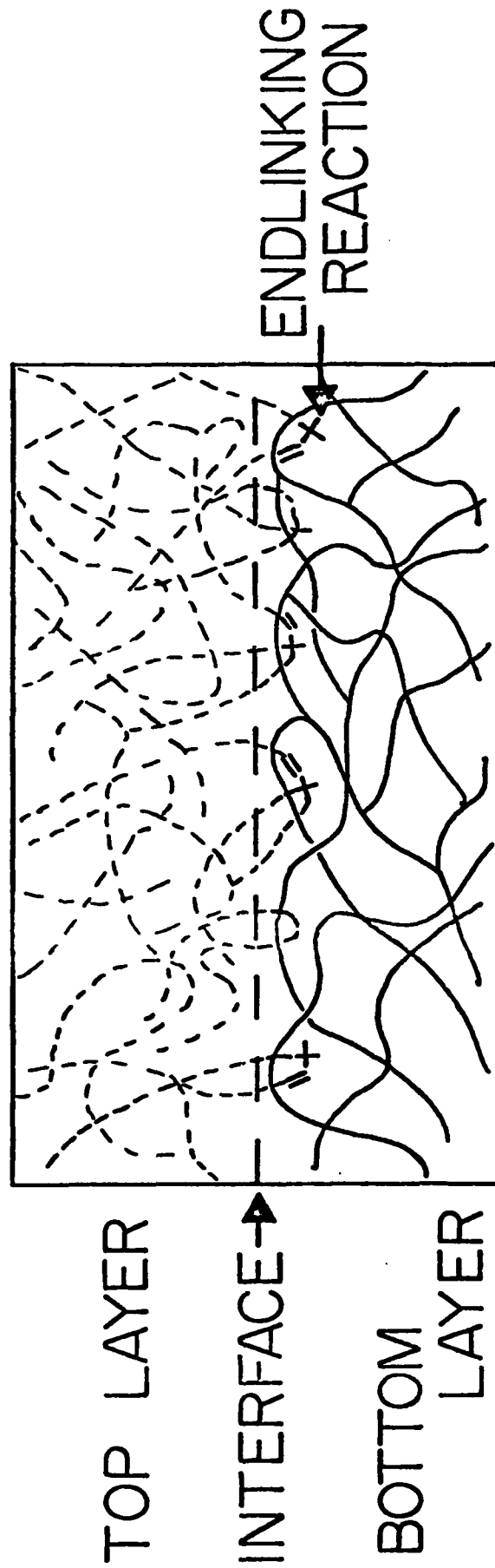


Figure 1. Sketch of the formation of macromolecular loops (permanent "entanglements") between an initially-gelled lower layer and an upper layer, applied and gelled later by an end-linking reaction.

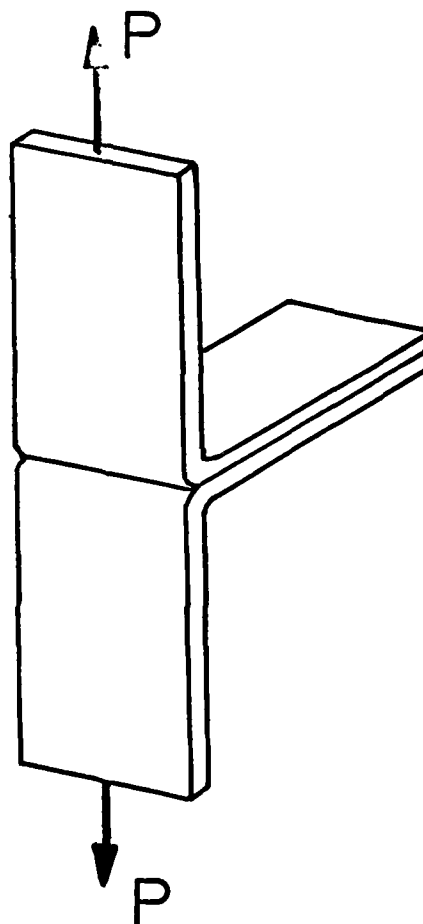


Figure 2. Method employed for measuring the work G_a of detachment. $G_a = 2P$, where \underline{P} is \underline{a} the peel force per unit width.

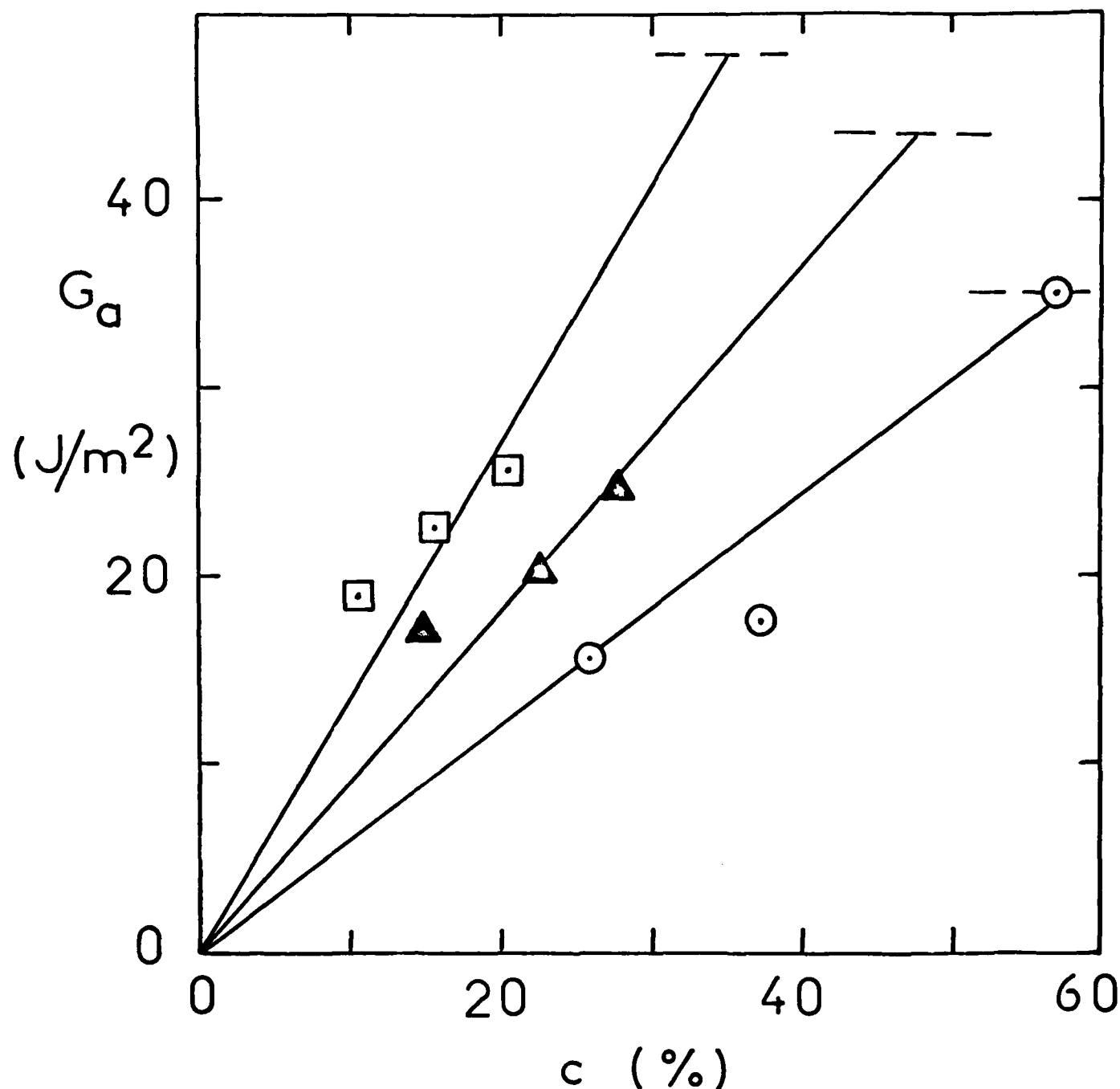


Figure 3. Relations between the work G_a of detachment of a PDMS layer and the concentration c of PDMS molecules absorbed by the lower layer before the upper layer was gelled. The horizontal broken lines denote the work G_c of fracture of the upper layer, after gelation. Molecular weight of PDMS used for the upper layer: 11,500, \circ ; 22,500, \blacktriangle ; 36,000, \square .

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